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## Preparation of Fibre Samples by the Kluft Method

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### Introduction

I have been asked by Roland Cox to attempt to repeat the method used by Kluft in PCT patent publication WO 97/24484 in order to prepare a fibre sample which can be tested for its effectiveness against *Aspergillus* species fungi and the bedmites which they support.

I have also been asked to prepare a fibre sample for the same purpose, again using the Kluft method but substituting Kluft's active agents by the fungicide, natamycin, used by Lebrun in United States patent No. 4,442,091.

### Kluft Sample A

#### (1) Materials

##### Fibres

Standard "Courtelle" acrylic fibres were used to provide comparability with "Amicor" fibres. The sample was a hank of blue dyed continuous filament tow having 200,000 filaments of 3.3 decitex and weighing approximately 25 gms.

##### Active Agents

Kluft is short on specifics and so we took his generally preferred active agents of a pyrethroid (as an acaricide) combined with a liquid mixture of equal parts by weight of 4-chloro-3-methyl phenol, orthophenyl phenol and a glycol ether. We chose di(ethylene glycol) ethyl ether for the glycol ether as one of the simplest members of this class. All components of this liquid mixture were 99% pure and were supplied by the Aldrich Chemical Company.

The pyrethroid chosen was the synthetic material, permethrin, because of its ready availability and effectiveness. Kluft's preference for a natural pyrethroid is based on considerations of biodegradability, which is not relevant for the purposes of these tests. Also, it may be questioned as a correct preference for a material that is supposed to have prolonged activity for the lifetime of the treated product, given that the natural material is liable to photolytic degradation. The permethrin was provided by Lifesystems Limited as an 11.4 % by weight solution formulated for application to textiles.

## **Binder**

Kluft's preferred binder is a perfluorinated acrylic compound. We chose a formulation widely-used as a water-repellent finish in the Textile Industry and sold by Ciba Speciality Chemicals plc under the brand "Oleophobol 7713". Alternative perfluorinated acrylic formulations were also tried in an attempt to overcome the problems referred to below; these were "Oleophobol SL" from Ciba Speciality Chemicals plc and "Nuva TP" from Clariant UK Ltd.

## **(2) Method**

### **Mode of Application**

Kluft sprays the agent/binder liquor onto the fibres during carding. This is not a suitable laboratory technique and is not critical. Simple immersion of the fibres in the liquor was used.

### **Concentration of Active Agents**

Kluft refers to concentration only when describing the preferred method illustrated by the drawing. He specifies a ratio of 0.4 to 0.6 % by weight of the biocide to the fibres being sprayed but does not identify what he means by the biocide.

We decided that, to be safe, we should aim to apply 0.5 % by weight based on the weight of the fibre, of the two phenols taken together, the phenols being what we believe to be the biocide/fungicide component of the mixture. In addition, again to be safe, we decided that we should apply the permethrin at a concentration of 0.4 % by weight of the permethrin on the weight of fibre.

### **Application Liquor**

Perfluorinated acrylic compounds like "Oleophobol 7713" are applied to textiles in the form of an aqueous emulsion. When the active agents were mixed into the "Oleophobol 7713" emulsion, they did not become dissolved or dispersed because the emulsion became destabilised and was unsuitable for application to the fibres. The same result was obtained with the alternative perfluorinated acrylic compositions tried. In view of this, it was decided that in order to obtain a treated sample at all, it was necessary to apply the active agents from solution in a solvent (acetone) as a first stage and then to apply the binder in a second stage. This was done.

In order to calculate the weight /volume concentration of the active agents in the acetone solvent required to give the desired pick-up of active agents on the fibres, the solvent pick-up of dry fibres was first measured. An equivalent weight of dry tow was immersed in acetone and then the saturated tow was manually squeezed to remove excess acetone before re-weighing. Pick-up was calculated as 75% volume/weight. The application liquor was prepared by dissolving in acetone ( AR Grade supplied by VWR International Ltd.), the requisite amounts of permethrin (23 gm. per 500 ml. acetone) and the liquid mixture of the two phenols and the glycol ether, each of which is added at 1.7 gm. per 500 ml. acetone.

## **Application**

The dry tow sample was immersed in the application liquor so as to become saturated, removed from the liquor, squeezed out by hand and then hung up to dry in ambient air for 90 minutes.

After drying, the treated tow sample was immersed to saturation in an aqueous emulsion of "Oleophobol 7713" (16.8 gm."Oleophobol 7713" in 500 ml. of water) to give a target application rate of the finish of 2.5% by weight on the weight of fibre. This is the application rate of Acordis's commercial oleophobic fibre.

The saturated tow was then wrung out by hand and hung up to dry in air for 20 minutes at room temperature. Finally, the tow was placed in an oven for 20 minutes at an air temperature of 110°C to dry and cure the applied perfluorinated finish.

## **Modified Kluft Sample B**

### **(1) Materials**

#### **Fibres and Binder**

These were the same as for the preparation of Sample A, with the exception that undyed fibre was used.

#### **Active Agent**

The agents used for the preparation of sample A were substituted by natamycin. This is a fungicide used predominantly in the Food Industry and is not readily available in pure form. A quantity was obtained as a 55:50 blend with lactose from AddFood Service GmbH.

### **(2) Method**

#### **Mode of Application**

The same immersion method as used to prepare Sample A was used.

#### **Concentration of Natamycin**

A target level of 0.5% by weight of natamycin on the weight of fibre was chosen.

#### **Application Liquor**

Natamycin is usually applied to food products by spraying it as a dry powder or as an aqueous dispersion. As natamycin did not dissolve in any commonly-available solvents tried, the two-stage application method used to prepare sample A was not suitable as a method in this case. It was decided, therefore, simply to disperse the natamycin directly in the aqueous emulsion of "Oleophobol 7713" to produce the application liquor.

### **Application**

The dry tow sample was immersed to saturation in the application liquor, removed from the liquor, squeezed out by hand and then dried and cured as in the preparation of sample A.

### **Resulting Samples A&B**

The treated Fibre Samples A&B were sealed in bags and given to Roland Cox to be sent for testing.

### **Distribution**

Roland Cox  
Jon Taylor  
Rex Smith  
Acrylics UK Archive (Elaine Topliss)